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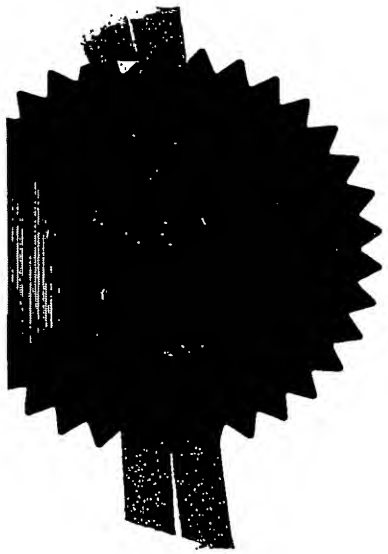

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07AUG03 E828321-1 002093
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SYNGENTA Limited
European Regional Centre
Priestley Road
Surrey Research Park, Guildford,
Surrey, GU2 7YH, United Kingdom

Patents ADP number *(if you know it)*

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If the applicant is a corporate body, give the country/state of its incorporation

UNITED KINGDOM

4. Title of the invention

FORMULATION

5. Name of your agent *(if you have one)*

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11. I/We request the grant of a patent on the basis of this application.

Signature Syngenta Limited C. Dowling Authorised Signatory Date 6 Aug 03

12. Name and daytime telephone number of person to contact in the United Kingdom

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FORMULATION

This invention relates to a formulation and in particular to a formulation of a solid suspended in an aqueous medium, for example an agrochemical suspension concentrate.

Solid, water-insoluble materials are often sold as suspensions in water. For agrochemical active ingredients, the term "suspension concentrate" is used to describe a suspension of a largely water insoluble active material in water. The suspension concentrate is intended to be diluted prior to application, for example by spraying. Such formulations avoid the need for solvents, have good handling characteristics, generally do not smell and are not flammable.

Dispersing aids are generally used to maintain the solids in suspension and adjuvants are commonly used to enhance the bioperformance (activity) of the agrochemical. Adjuvants can vary in complexity from simple surfactants to multi-component blends of oils. Such adjuvants may be added to agricultural spray tanks separately from the agrochemical formulation but it is more convenient to provide a formulation where all necessary dispersing aids and adjuvants are incorporated directly in ("built-in") the suspension concentrate. This ensures that the farmer will use the correct adjuvant, and also controls the ratio of adjuvant to active material. Oil-based adjuvants are particularly useful in enhancing the bioperformance of an agrochemical but are also inherently difficult to build-in to an aqueous concentrate at desirably high concentration. As used herein, the term "oil-based adjuvant" means a liquid adjuvant or mixture of adjuvants that is substantially insoluble in the aqueous medium in which the solid agrochemical is suspended. Suspension concentrates can be formulated with surfactants and oil blends but this often leads to problems of compatibility, particularly since the suspension concentrate is required to be physically stable over extended periods of time and under the extremes of temperature encountered during commercial use. Stability problems may include flocculation, crystal growth of the dispersed solid, creaming of the adjuvant/surfactant system, phase separation and chemical decomposition.

It is desirable therefore to provide a physically stable suspension concentrate that contains a built-in bioperformance enhancing oil-based adjuvant at high loading.

According to the present invention there is provided an aqueous agrochemical suspension concentrate characterised in that it comprises an oil-based adjuvant and a hydrotrope capable of solubilising said adjuvant in the aqueous phase.

A hydrotrope is a substance that, at high concentrations, enhances the solubility of non-polar compounds (oils) in water. Solubilisation of oils by hydrotropes is characterised by the relatively high concentrations of the hydrotropes needed and the larger amount of oil solubilised compared with that observed for conventional micellar surfactants. Examples of hydrotropes which may be used in the present invention include anionic benzoates, anionic benzosulphonates, anionic phosphates and phosphonates, anionic benzophosphates, alkylarylphosphates and phosphonates, neutral phenols such as catechol and resorcinol, aliphatic glycolsulfates, alicyclic bile salts, aliphatic carboxylates, aromatic carboxylates, naphthalene sulphonates, alkynaphthalene sulphonates, polymeric naphthalene sulphonates and their copolymers, alkyl aryl sulphonates and carboxylates and their polymers and copolymers, naphthalene and alkylnaphthalene phosphates and phosphonates and their polmers and copolymers, glycol and glycerol ethers and the amino acid proline. In general a given hydrotrope will solubilise a specific group of oils or oil blends.

As examples of oil-based agrochemical adjuvants suitable for use in combination with the above hydrotropes there may be mentioned seed oils, methylated seed oils, triglycerides of fatty acids and fatty amines, methyl esters of fatty acids and fatty amines, mineral oils which can be linear, branched or mixtures of linear and branched, aromatic oils, fatty alcohols, fatty acids, fatty amines, aliphatic alcohols, aliphatic amines, aliphatic esters, aliphatic carboxylic acids, aliphatic ketones, aliphatic aldehydes, aliphatic amides, aromatic carboxylic acids, aromatic alcohols and phenols, aromatic ketones, aromatic aldehydes, aromatic amines or anilines or anilides, aromatic amides, natural products such as terpenes, sesquiterpenes and diterpenes, alkyl or aryl or alkylaryl phosphates and phosphonates. Included also are halogenated variants of the forementioned oils. Similarly ethoxylated variants of the alcohols, amines and acids mentioned are also suitable providing the degree of ethoxylation is not too long, for example having an average degree of ethoxylation below 4. Brij 92, oleyl alcohol ethoxylate with an average of 2 moles of ethoxylate is an example of a suitable ethoxylated fatty alcohol and Ethomeen S12 is an example of a short chain ethoxylated fatty amine. Silicone oils are also suitable. Oil blends may also be used, for example Turbocharge (Turbocharge is a tradename of Syngenta Limited), which is a proprietary blend of oils and short chain ethoxylates. Other commercially available blends of oils and short chain ethoxylates

include Merge, Dash, BreakThru 464 and Agridex. The commercial product Agral 90 is ablend of ethoxylated nonylphenols.

As examples of preferred combinations of hydrotrope and oil-based adjuvants suitable for use to improve the bioperformance of agrochemicals, there may be mentioned:

(a) Benzosulphonate hydrotropes such as ammonium cumene sulphonate and ammonium xylene sulphonate in combination with fatty alcohols, fatty acids or fatty amines and simple derivatives thereof such as methyl esters and adjuvant oils derived from plant terpenes.

(b) Anionic alkylaryl carboxylatehydrotropes such as the potassium salt of 5(6)-carboxy-4-hexyl-2-cyclohexene-1 octanoic acid (commercially available under the trade name WESTVACCO H240) in combination with fatty alcohols, fatty acids or fatty amines and simple derivatives thereof such as methyl esters and short chain ethoxylates, especially when these materials are also blended with linear or branched mineral oils.

As preferred examples of combinations of type (a) above there may be mentioned the hydrotrope ammonium cumene sulphonate in combination with oleyl alchol or the plant terpene phytol or the plant terpene geraniol and the hydrotrope ammonium xylene sulphonate in combinaton with the plant terpene geraniol. As preferred combinations of type (b) above there may be mentioned the hydrotrope 5(6)-carboxy-4-hexyl-2-cyclohexene-1 octanoic acid in combination with oleyl alcohol or the commercially available adjuvant Turbocharge. We have found that for agrochemicals such as selective herbicides, insecticides and fungicides that are not intended to harm the target plant, the built-in combination of the oil-based adjuvant and the hydrotrope may often be less phytotoxic to target plants than the corresponding tank-mixed adjuvant system, whilst delivering equivalent bioperformance.

It is to be understood that the hydrotrope solubilises the oil-based adjuvant into the aqueous medium in which the agrochemical is suspended in the suspension concentrate of the present invention. The aqueous medium need not necessarily be a true solution in the physical sense but will appear to be a single phase to the eye and under the microscope and will remain as such over extended storage periods, and in many instances essentially indefinitely. It is a further advantage of the hydrotrope solubilised systems of the present invention, not only that relatively high levels of oil-based adjuvant may be

built-in but also that the resultant composition has a relatively low viscosity. Such compositions are poured from a container without difficulty and without leaving excessive residues in the container. The composition is readily diluted in the spray tank and any residues remaining in an empty container are simply and easily rinsed out. The compositions of the invention are typically Newtonian fluids with viscosity less than 1000 cP at room temperature.

The present invention does not depend critically on the nature of the agrochemical active ingredient present in the suspension concentrate. Those skilled in the art will be well aware of the criteria for a given agrochemical to be suitable for formulation as a suspension concentrate. As examples of herbicides suitable for formulation as a suspension concentrate there may be mentioned mesotrione, fomesafen, tralkoxydim, napropamide, amitraz, propanil, pyrimethanil, dicloran, tecnazene, toclofos methyl, flamprop M, 2,4-D, MCPA, mecoprop, clodinafop-propargyl, cyhalofop-butyl, diclofop methyl, haloxyfop, quizalofop-P, indol-3-ylacetic acid, 1-naphthylacetic acid, isoxaben, tebutam, chlorthal dimethyl, benomyl, benfuresate, dicamba, dichlobenil, benazolin, triazoxide, fluazuron, teflubenzuron, phenmedipham, acetochlor, alachlor, metolachlor, pretilachlor, thenylchlor, alloxydim, butroxydim, clethodim, cyclodim, sethoxydim, tepraloxydim, pendimethalin, dinoterb, bifenox, oxyfluorfen, acifluorfen, fluoroglycofen-ethyl, bromoxynil, ioxynil, imazamethabenz-methyl, imazapyr, imazaquin, imazethapyr, imazapic, imazamox, flumioxazin, flumiclorac-pentyl, picloram, amodosulfuron, chlorsulfuron, nicosulfuron, rimsulfuron, triasulfuron, triallate, pebulate, prosulfocarb, molinate, atrazine, simazine, cyanazine, ametryn, prometryn, terbuthylazine, terbutryn, sulcotrione, isoproturon, linuron, fenuron, chlorotoluron, metoxuron, fungicides such as azoxystrobin, trifloxystrobin, kresoxim methyl, famoxadone, metominostrobin and picoxystrobin, carbendazim, thiabendazole, dimethomorph, vinclozolin, iprodione, dithiocarbamate, imazalil, prochloraz, fluquinconazole, epoxiconazole, flutriafol, azaconazole, bitertanol, bromuconazole, cyproconazole, difenoconazole, hexaconazole, paclobutrazole, propiconazole, tebuconazole, triadimefon, trtiticonazole, fenpropimorph, tridemorph, fenpropidin, mancozeb, metiram, chlorothalonil, thiram, ziram, captafol, captan, folpet, fluazinam, flutolanil, carboxin, metalaxyl, bupirimate, ethirimol, dimoxystrobin, fluoxastrobin, orysastrobin, metominostrobin and prothioconazole.

As examples of fungicides suitable for formulation as a suspension concentrate there may be mentioned azoxystrobin, trifloxystrobin, kresoxim methyl, famoxadone, metominostrobin and picoxystrobin, cyprodanil, carbendazim, thiabendazole, dimethomorph, vinclozolin, iprodione, dithiocarbamate, imazalil, prochloraz, fluquinconazole, epoxiconazole, flutriafol, azaconazole, bitertanol, bromuconazole, cyproconazole, difenoconazole, hexaconazole, paclobutrazole, propiconazole, tebuconazole, triadimefon, trtiticonazole, fenpropimorph, tridemorph, fenpropidin, mancozeb, metiram, chlorothalonil, thiram, ziram, captafol, captan, folpet, fluazinam, flutolanil, carboxin, metalaxyl, bupirimate, ethirimol, dimoxystrobin, fluoxastrobin, orysastrobin, metominostrobin and prothioconazole

As examples of insecticides suitable for formulation as a suspension concentrate there may be mentioned thiamethoxam, imidacloprid, acetamiprid, clothianidin, dinotefuran, nitenpyram, fipronil, abamectin, emamectin, bendiocarb, carbaryl, fenoxycarb, isoprocarb, pirimicarb, propoxur, xylylcarb, asulam, chlorpropham, endosulfan, heptachlor, tebufenozide, bensultap, diethofencarb, pirimiphos methyl, aldicarb, methomyl, cyprmethrin, bioallethrin, deltamethrin, lambda cyhalothrin, cyhalothrin, cyfluthrin, fenvalerate, imiprothrin, permethrin, halfenprox.

The foregoing lists are not intended to be exhaustive and other examples will occur to those skilled in the art.

The suspension concentrate of the present invention may also incorporate one or more surfactants or dispersing agents to assist the suspension of the solid agrochemical in the aqueous medium (dispersant system). The dispersant system will not generally contribute to enhancement of the bioperformance of the agrochemical and is present primarily to assist in maintaining the dispersed solid agrochemical in suspension. Conversely the oil-based adjuvant will not generally assist directly in maintaining the agrochemical in suspension. Many individual dispersants and mixtures thereof suitable for forming a dispersant system for a suspension concentrate are known to those skilled in the art and a very wide range of choices is available. Typical dispersants that may be used to form a dispersant system include copolymers of ethylene oxide and propylene oxide, aryl and alkyl aryl sulphonate copolymers with formaldehyde such as naphthalene sulphonate formaldehyde copolymers, salts of the copolymers of acrylic acid with diisobutylene or ethylene oxide or styrene or vinyl pyrrolidone, salts of copolymers of styrene sulphate with ethylene oxide or diisobutylene or vinyl pyrrolidone or propylene

oxide, tristyrylphenol type dispersants where the phenol has been ethoxylated and optionally sulphonated or phosphated, alkylphenol ethoxylates, polyvinyl alcohol and substituted or sulphated polyvinyl alcohols, polyvinyl pyrrolidone and its copolymers.

The agrochemical is conventionally present in the suspension concentrate at a concentration between 5 and 60 % and typically of about 10 to 35% by weight. The hydrotrope is suitably present in the suspension concentrate at a concentration of from 5 to 50 % and typically from about 15 to 30% by weight. The oil-based adjuvant is suitably present at a concentration of from 5 to 60 % and typically from 5 to 40% by weight. The dispersant system is typically present at a concentration of total dispersant of from 0 to 40 % and preferably from 0 to 20% by weight. The dispersant system may comprise a mixture of dispersants. A typical example of a mixture of dispersants includes a copolymer of ethylene oxide and propylene oxide, such as Atlox 4894 or Atlox 4896, in a quantity from 0 to 20 % for example from 1 to 8 % w/w combined with a further dispersant such as Atlox 10/5 or Brij 96 in a concentration of 0 to 20 %, for example from 1 to 9 % w/w.

The ratio of oil-based adjuvant to hydrotrope suitably varies from 1:10 to 10:1, for example from 1:3 to 3:1. The suspension concentrate may be made using conventional techniques. Typically in commercial practice, the solid herbicide is milled in water until the desired particle size is reached. The particle size is typically from 0.5 to 15 microns, for example from 1 to 5 microns volume median diameter. The dispersant system is generally added before milling so that it is present during the milling process. The stage at which the hydrotrope and oil-based adjuvant are added is not critical. It is generally convenient to add the hydrotrope together with the oil to the aqueous system before, during or after milling.

According to another aspect of the present invention there is provided a process for the manufacture of a suspension concentrate which comprises milling a solid agrochemical in water, optionally in the presence of a dispersant system, characterised in that there is incorporated in the composition a hydrotrope and an oil-based adjuvant for the agrochemical.

The invention is illustrated by the following Examples in which all parts and percentages are by weight unless otherwise stated.

The composition of the products used in the Examples was as follows: -

Morwet D425 – A commercially available anionic naphthalene sulphonate formaldehyde condensate copolymer, sold by Crompton Corporation

Synperonic 10/5 – A commercially available surfactant comprising a short chain branched alcohol with five moles of ethylene oxide. Sold by Uniqema Ltd

Atlox 4896 – A copolymer condensate of ethylene and propylene oxide, sold by Uniqema Ltd.

Atlox 4913 - A copolymer condensate of ethylene oxide with methyl methacrylate, sold by Uniqema Ltd.

Atlox 4894 - A copolymer condensate of ethylene and propylene oxide, sold by Uniqema Ltd.

Brij 96 – A fatty alcohol condensed with an average of 10 moles of ethylene oxide. Sold by Uniqema Ltd

Eltesol AC 60 – Ammonium cumene sulphonate supplied by Albright and Wilson Ltd

EXAMPLE 1

The herbicide tralkoxydim (10 %) having a particle size of about 50 microns, Morwet D425 (8 %), Westvacco H240 (29 %), Turbocharge (30 %), Synperonic 10/5 (6 %) and water (17 %) to a total weight of 5 g was added to a glass vial. The Westvacco H240 was the hydrotrope and the Turbocharge was the oil-based adjuvant. Morwet D425 and Synperonic 10/5 together formed the dispersant system. The mixture was gently swirled and sheared for one minute using an Ystral mixer, which reduced the particle size of the tralkoxydim to a volume mean of 25 microns. An equal volume of No 4 zirconia beads was added to the vial which was then shaken for 30 minutes in a laboratory shaker.

The sample could be both poured and pipetted easily and this was taken as an indication that the viscosity was satisfactory (viscosity test). The sample dispersed readily in water without agitation, at a dilution of 1 % and a standing time of one minute (dilution test).

The sample also passed a standard flocculation test. In this test a 1 % dilution of the sample was made using CIPAC standard hard water C. The sample was inverted to ensure homogeneity and was left to stand for one hour. After this time it was examined by microscope to observe any signs of flocculation. If there were no signs of flocculation the sample met the flocculation test.

EXAMPLES 2 to 5

The procedure described in example 1 was repeated for the samples shown in Table 1. These samples all contained the commercial herbicide tralkoxydim with the commercial tank mix adjuvant Turbocharge. All samples passed the viscosity, dilution and flocculation tests.

Table 1 – Composition in % by weight

Example No	Tralkoxydim	Morwet D425	Westvacco H240	Turbocharge	Synperonic 10/5	Water
2	12	4	25	38	0	21
3	16	5	27	25	0	27
4	15	5	25	25	5	25
5	16	2	25	22	0	35

EXAMPLES 6 TO 11

The compositions listed in Table 2 were prepared using the procedure of Example 1. In each case however the dispersant Morwet D425 has been replaced with Atlox 4894. All samples passed the viscosity, dilution and flocculation tests.

Table 2 – Composition in % by weight

Example No	Tralkoxydim	Westvacco H240	Turbocharge	Synperonic 10/5	Atlox 4894	Water
6	10	25	17	9	8	31
7	10	30	25	8	6	21
8	10	20	30	3	8	29
9	13	19	26	3	8	31
10	10	22	11	5	0	52
11	20	31	20	7	8	14

EXAMPLES 12 TO 15

The compositions listed in Table 3 were prepared using the procedure of Example1. The Examples illustrate the use of different dispersant systems. All samples

passed the viscosity, dilution and flocculation tests. In Table 3, D1 is Atlox 4896, D2is Atlox 4913, D3 is Atlox 4894 and D4 is Morwet D425

Table 3 – Composition in % by weight

Example No	Tralkoxydim	Westvacco H240	Turbocharge	Synperonic 10/5	Water	D1	D2	D3	D4
12	20	31	20	7	14	8	0	0	0
13	20	31	20	7	14	4	4	0	0
14	20	31	20	7	14	0	0	4	4
15	20	31	20	7	14	0	0	8	0

EXAMPLES 16 AND 17

The compositions listed in Table 4 were prepared using the procedure of Example1. The Examples illustrate the use of different dispersant systems. All samples passed the viscosity, dilution and flocculation tests. In Table 4, C1 is Synperonic 10/5, C2 is Brij 96.

Table 4 – Composition in % by weight

Example No	Tralkoxydim	Morwet D425	Westvacco H240	Turbocharge	Water	C1	C2
16	15	5	25	25	25	0	5
17	15	5	25	25	25	5	0

EXAMPLES 18 AND 19

The compositions listed in Table 5 were prepared using the procedure of Example 1 but used the commercial herbicide diuron in place of tralkoxydim. All samples passed the viscosity, dilution and flocculation tests.

Table 5 – Composition in % by weight

Example No	Diuron	Atlox 4894	Westvacco H240	Turbocharge	Synperonic 10/5	Water
18	13	8	19	26	3	31
19	10	8	20	30	3	29

EXAMPLES 20 TO 23

The compositions listed in Table 6 were prepared using the procedure of Example1 but used the commercial fungicide picoxystrobin in place of tralkoxydim. All samples passed the viscosity, dilution and flocculation tests.

Table 6 – Composition in % by weight

Example No	Picoxystrobin	Atlox 4894	Westvacco H240	Turbocharge	Synperonic 10/5	Water
20	21	6	27	19	4	23
21	24	5	27	17	3	24
22	19	8	26	17	3	27
23	30	8	31	17	3	11

EXAMPLE 24

This example illustrates the formation of a hydrotrope formulation containing the oil oleyl alcohol with the hydrotrope ammonium cumene sulphonate.

Tralkoxydim (10 %), Atlox 4894 (8 %), Eltesol AC 60 (13 %), Oleyl alcohol (30 %), Synperonic 10/5 (3 %) and water (36 %) to a total weight of 5 g was added to a glass vial. The mixture was gently swirled and sheared for one minute using an Ystral mixer. An equal volume of no 4 zirconia beads was added to the vial which was then shaken for 30 minutes in a laboratory shaker.

The resulting sample passed the viscosity, dilution and flocculation tests.

EXAMPLE 25

Three hydrotrope formulations were prepared using the method outlined in Example 1. Table 7 shows the ingredients that were used.

Table 7 – Composition in % by weight

Composition	Tralkoxydim	Westvacco H240	Atlox 4894	Turbocharge	Synperonic 10/5	Water
H 1	20	31	8	20	7	14
H 2	13	19	8	26	3	31
H 3	10	20	8	30	3	29

These samples passed the viscosity, dilution and flocculation tests, and were used for biological testing.

A sample of a commercial tralkoxydim suspension concentrate available under the tradename 'Achieve 25 SC' was used as a reference material. The strength of the suspension concentrate was 25 % w/w. The reference material was tank mixed with commercial Turbocharge at two rates, these being 0.2 % and 0.5 % v/v of the spray tank volume. The ratio by weight of Turbocharge to tralkoxydim in the compositions is given below in Table 11 at an application rate of 50 g/ha. Plant species (weeds against which tralkoxydim is effective) were grown and tested under glasshouse conditions. *Alopecuius myosuroides* (ALOMI), *Avena fatua* (AVEFA), *Echinochloa crus-galli* (ECHCG) and *Lolium rigidum* (LOLRI) were grown to the 2.3 leaf stage and *Setaria viridis* (SETVI) to stage 3.3, and were sprayed with 100 litres/hectare of spray solution using a laboratory track sprayer. The pesticide application rate was 50 g/ha. Tralkoxydim is a selective herbicide for use on wheat and barley and phytotoxicity against these species is therefore undesirable. The wheat species *Triticum aestivum* (TRZAS) cultivar 'Barrie', and the barley variant *Hordeum vulgaris* (HORVU) 'Bonanza' were sprayed at rates of 50, 100, 200 and 400 g/ha. The percentage crop damage was assessed at 11 and 22 days after application, and the weed control at 22 days after application.

Table 8 shows the phytotoxicity of each formulation scored as percentage leaf damage on two crop species 11 days after treatment. The score represents the mean value taken from all of the rates, with three replicated of each rate.

Table 8 Leaf Damage 11 DAT %

Composition	TRZAS 'Barrie'	HORVU 'Bonanza'
H1	5	2
H2	3	4
H3	10	2
Achieve SC' + 0.2 % Turbocharge	13	7
Achieve SC' + 0.5 % Turbocharge	15	12

Table 9 shows the percentage kill for each formulation on five weed species. The top rate of tralkoxydim (50 g/ha) was used and results are the mean of three replicates. At this application rate the ratio of Turbocharge to tralkoxydim for the five formulations is shown in Table 10.

Table 9 - % Kill against Weed Species

Formulation	ALOMY	AVEFA	LOLRI	SETVI
H 1	75.0	80.0	63.3	61.7
H 2	88.3	85.0	43.3	46.7
H 3	86.7	93.3	68.3	73.3
Achieve SC' + 0.2 % Turbocharge	86.7	86.7	68.3	65.0
Achieve SC' + 0.5 % Turbocharge	86.7	90.0	83.3	66.7

Table 10 – Ratio of Turbocharge to Tralkoxydim

Formulation	Ratio Turbocharge/Tralkoxydim
H1	1
H2	2
H3	3
Achieve SC' + 0.2 % Turbocharge	3.5
Achieve SC' + 0.5 % Turbocharge	8.75

The biological efficacy of each formulation as a weed killer is statistically the same. As the hydrotrope formulations all contained less Turbocharge relative to the tralkoxydim than the tank mixed formulations they were therefore as good as, or better than, the tank mixed formulations, while at the same time being less phytotoxic to crop species.

CLAIMS

1. An aqueous agrochemical suspension concentrate characterised in that it comprises an oil-based adjuvant and a hydrotrope capable of solubilising said adjuvant in the aqueous phase.

ABSTRACT

An aqueous agrochemical suspension concentrate comprises an oil-based adjuvant and a hydrotrope capable of solubilising said adjuvant in the aqueous phase.